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RESEARCH ON MATERIALS FOR HIGH POWER LASER WINDOWS

N. J. Grant, et al

Massachusetts Institute of Technology

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RESEARCH ON MATERIALS FOR HIGH POWER LASER WINDOWS

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13. ABSTRACT

The mechanical properties of polycrystalline alkali halides have been investigated. The effects of texture, surface preparation, and coatings have been shown to significantly effect the stress-strain characteristics; samples with strengths over 5000 psi and with considerable ductility have been prepared. The plane strain fracture toughness has been measured and indicated a factor of 3.5 improvement in the resistance of crack propagation. The K_{IC} values for polycrystalline KCl were 943 psi·in$^{1/2}$ compared to 272 psi·in$^{1/2}$ for the single crystal. Flaw detection, fractography, and laser damage studies have also been initiated. Studies of thin film coatings of Ge, MgF_2, and NaF on polycrystalline KCl have continued. Double salt crystals of barium chloride-potassium chloride were grown for optical absorption measurements. The IR absorption edge occurs at 14μm.

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Principal Investigator
N.J. Grant
(617) 253-5638

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Massachusetts Institute
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AFCRL Project Scientist
Harold Posen
(617) 861-3532

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CONTENTS

FABRICATION OF POLYCRYSTALLINE KCl WINDOWS

The techniques reported in the last quarterly report for the formation of polycrystalline windows from alkali halide single crystals by hot-deformation and recrystallization have been further investigated. Besides the production of many large samples (2 inch diameter by 1/4 inch thick) for mechanical properties studies, investigation of the plastic flow characteristics during deformation, of the recrystallization kinetics, and of the effects of texture has begun.

Flow characteristics. The hot-deformation temperature range that we have found most convenient for controlling the grain size, minimizing residual stresses, and eliminating rim cracks is 250 to 300°C. Since the deformation is performed in a cylindrical die, the uniaxial loading at the beginning of deformation has a hydrostatic component at the final conditions because of the constraining action of the die walls. To determine whether purely plastic flow was operative at these deformation temperatures an experiment was performed to document the flow behavior.

A cylindrically shaped single crystal (1 inch diameter by 1/2 inch thick) with the (100) direction parallel to the cylindrical axis was cleaved at the half thickness (Figure 1). Four 1 mm diameter holes were drilled in one surface at the center and three at radially random points. The cleaved crystal surfaces were precisely re-joined and the proper orientation maintained by placing droplets of glue on the outer cylindrical surface. The placement of the four holes and their orientation to the crystallographic axes were noted prior to placing the specimen in the vacuum hot-forging facility. Hot-deformation was carried out at 275°C with a 4 to 1 reduction in height and with a vacuum of 5×10^{-5} torr. The temperature was maintained prior to deformation for about 1 hour to insure evacuation of the four holes.

Examination of the polycrystalline sample after the hot-work, recrystallization process indicated that the mass flow during deformation had at least cubic symmetry. In Figure 1, the position of the four holes was observable as very faint circles.

Texture and orientation. More than fifty 2 inch diameter KCl discs have been prepared by high temperature deformation and recrystallization. The orientation of the single crystals had the (100) direction parallel to the deformation direction. As shown in Figure 2, additional specimens have been prepared in which the (111) and (110) crystallographic directions were the pressing directions; Figure 2 also shows a 1 inch KCl-KBr alloy pressed in the (100) direction. The previously described methods[1] were used with the (111) and (110) orientations. The polycrystalline billets had average grain sizes as small as 6μ for the discs pressed with a (110) orientation and 10μ for the (111) orientation. An etched micrograph of one (110) sample is shown in Figure 3. Besides the hot-pressed samples, an extrusion technique is being developed to impart other texture to the polycrystalline material. A vacuum billet casting apparatus has been constructed for the production of large billets for extrusion.

Laue patterns of the polycrystalline billets show extensive arcs (20-60°) indicating that significant texture remains in the fine grained billets. The pole figures reported by Kulin et.al.[2] for similarly prepared polycrystalline KCl have confirmed this lack of random grain orientation. Samples of the (100) pressed material were supplied to AFCRL for determination of the degree of grain to grain misorientation. On a specimen which had been annealed to produce large grains, the orientation of each grain was determined from Laue patterns. The grain to grain misorientation of (100) poles or 14 grains varied from a few degrees to about 20°.* A fine grained sample was replicated to show slip traces in transmission electron micrographs. The slip lines appear to have 150 Å spacings while an apparent grain misorientation determined by the angle between the slip lines of 11° was observed.+

Additional samples have been hot-worked to yield another type of textural effect. The cleaved crystals shown schematically in Figure 1 were

* Xray measurements made by Jane Bruce of Air Force Cambridge Research Labs

\+ Electron microscope work performed by Joseph Kolmer of Air Force Cambridge Research Labs misoriented by 45° (in the plane of the disc) and subsequently hot-deformed to 2 inch discs. Another crystal was cleaved into thirds and each was misoriented by 30°. These sandwiched orientations and others with {110} and (111) directions parallel to the direction of deformation will be tested to determine maximum bend strength configurations.

Recrystallization Kinetics. Very little is known about the recrystallication kinetics or grain growth kinetics of polycrystalline halides. The only comprehensive study was made by Mueller[3] in 1935 on NaCl crystals. A similar study on KCl has been initiated. Small cubes (5 mm on an edge) of KCl are deformed at different temperatures and different total strains in a small furnace arrangement mounted on an Instron test machine. The strained crystals are immediately quenched to room temperature. The recrystallization velocity and nucleation time are measured in a hot-stage microscope. The orientation of the new crystal relative to the original orientation can also be tabulated.

YIELD STRENGTH AND FRACTURE STRENGTH

It was previously demonstrated[1] that the optical transimission characteristics of the polycrystalline KCl is not degraded relative to the single crystal properties; yet there were large increases in the mechanical properties. To further document these increases and to determine the added effects of particular textures, more tests have been carried out.

Texture Effects. Compression tests have been made along the <111> direction in KCl single crystals at room temperature. This is the strongest orientation in single crystals and in perfectly textured polycrystals slipping on {110} <110> systems, the reason being that the Schmid factor on such systems is zero for loading exactly along <111>. The specimens were prepared from Harshaw single crystals, after cleaving along {100}, by mounting on a goniometer, reorienting properly, and polishing with a water soaked felt wheel. Final height and diameter were about the same so that "friction-hill effects" could not have seriously influenced measured strengths.

An average compressive yield stress of 1650 psi was obtained in the first experiments on crystals of somewhat rough and irregular lateral faces. The measured strength was nearly doubled in a second set of tests by refinements in preparation and handling. The refinements included polishing all sides of the sample and rounding over edges to eliminate surface cracks and other sources of stress concentration. Specimens were never touched directly and were always stored in an evacuated dessicator. Both lead and Teflon films were used for lubricants and pressure distributing agents. Failure was defined by the initial appearance of cracks, which always started at the edges of the loaded faces where they are normally found in brittle solids. Analysis after test showed cracking on the {110} plane in the <110> direction, which is the slip system that should not have been activated. Under such high axial pressure, however, a misalignment of about a degree would be enough for a resolved shear stress of 75 psi on these systems. Whether there was that much misalignment is not known with certainty; the problem will be dealt with more explicitly in future tests. One sample was loaded until the initial cracks penetrated well into the interior. The total strain, still without complete fracture, was about 16% and the final stress was nearly 10,000 psi. There was significant barreling and every indication of plastic deformation along with the cracking.

Other experiments are being planned with further improvements in specimen design. Samples now in preparation are to be even more nearly cylindrical and free of crack-initiation sites. The measured strengths are probably still specimen-design rather than materials limited, and it is important to minimize that influence. Strain will also be more accurately measured in tests to come with wire-resistance strain gauges attached to the side of the crystal. Compression experiments are also planned along the <110> direction.

These compression experiments are designed to evaluate the theoretical texture hardening potential in polycrystalline KCl. There is no obvious reason why much if not all of that potential could not be superposed on a base from grain-size hardening, provided the appropriate texture is imparted to the polycrystals

Strength of Polycrystalline Halides. Four point bend tests were carried out as previously described[1] on samples cut from 2 inch diameter discs. Figure 4 contains the comparative data for several different samples. Although there is a small amount of yield which gives an apparent elastic limit, all of the polycrystalline samples fail by brittle fracture thus the values represent the modulus of ruputre.

The most significant strengthening effects were observed for samples cut from billets which were hot-worked in the (110) direction. Modulus of ruptures of nearly [illegible]00 psi were observed. The (111) oriented billets showed the least strength. These observations are somewhat tentative until samples of comparative grain size are tested.

Only large grained polycrystalline KCl-KBr alloy has been produced to date due to the high processing temperatures required. For a billet with a grain size of 300μm, the fracture strength was 2460 psi compared to 1350 psi for single crystals of the same alloy. This high value is encouraging especially for such a large grained specimen. If one assumes a Petch relation, as has been demonstrated for polycrystalline KCl,[4] and extrapolates to a 50μm grain size a fracture strength of 5000 psi is predicted.

The Vickers hardness of the polycrystalline samples was measured for the specimens shown in Figure 4 and are given in Table I. The hardness for the polycrystalline samples shows the same variations with respect to pre-hot work orientation as the single crystal values.

Surface preparation. It is well known that the mechanical properties of brittle materials are strongly dependent upon the surface preparation. The presence of surface flaws due to sample preparation methods reduces the observed strengths below the intrinsic values and also causes a distribution in the measured values according to the variation in the distribution and size of flaws from sample to sample. As previously reported, although extreme care was taken to prepare polycrystalline KCl samples for four-point bend measurements, scatter in the measured values was observed as well as observations of the common occurence of fracture initiation during testing at a location not at maximum stress. Thus, procedures have been started to carefully polish and protect the sample surfaces.

Bend specimens were cut from polycrystalline billets formed from crystals oriented in the (110) and (111) direction. Rectangular beams were carefully polished as previously described. After polishing, they were dipped into distilled water for 30-40 seconds to chemically etch the damaged surface layers. They were immediately dipped into pure ethanol to remove the water and quickly dried. Each dried sample was then coated with a thin layer of spray lacquer and left to dry overnight. The lacquer, Krylon*, provides a thin polymeric protective coating which protects the surface from scratches and moisture. It is also plastic and deformable such that it provides a coherent protective layer even during bend tests.

The first bend test results confirmed a notion of the deleterious effects of surface flaws. Figure 5 shows the data for samples similar to those tested in Figure 4 which had no special surface preparation. Two important differences in the stress-deflection diagrams are worth noting. Firstly, the curves indicate a definite yield point followed by a significant degree of plastic flow. Secondly, instead of a large degree of scatter in the brittle fracture results as shown schematically in Figure 5, there is less variation in the final fracture stresses which occur after significant strain.

These results imply that surface coatings of laser window materials are not only important to enhance optical transmittance, i.e. as anti-reflective coatings and for protection against moisture attack resulting in optical losses at the surface, but also that the mechanical properties of the window system (base material and coating) are improved to the intrinsic properties of the base material. In addition, the very nature of designing a system based on brittle materials requires special caution for the probability of catastropic failure. Yet polycrystalline KCl when properly prepared and protected from surface flaws appears to have sufficient ductility to avoid many probable failures. The optical transmission may degrade or a lensing effect may result from plastic yielding (for example, for loads greater than ≈3800 psi for (110) material in Figure 5); but the system will not undergo catastropic failure.

* Krylon No. 1302 manufactured by Borden, Inc.

More tests are planned for material coated in this manner especially with regard to fatigue characteristics. Although lacquer type coatings are of little value for transmission of CO_2 laser wavelengths, they are convenient to use for room temperature mechanical property studies.

PLANE STRAIN FRACTURE TOUGHNESS

Ten fracture toughness tests have been conducted in room air (percent relative humidit from 30 to 60%) and at room temperature. In testing KCl crystals the following problems should be kept in mind:

1) Their extreme notch sensitivity, especially the single crystals, adds to the critical nature of the initial notch geometry for the consistency of the results. Induction of a sharp initial crack by fatigue is made difficult not only by the lack of such equipment at present but also by the fact that such cracks may rapidly propagate across the entire crystal.

2) Their hygroscopic tendency, especially that of the polycrystals, requires their protection against moisture up to the moment of testing.

3) Existing ASTM Standards (E399-70T) are developed strictly for metals. The use of the ASTM criterion for obtaining the minimum thickness requirements result in a thickness of the single crystals of about 1 inch. The ASTM requirements were satisfied, however, by the polycrystalline thicknesses available (0.25 inch).

Thus in choosing the specimen geometry we adhered to the ASTM requirements only as much as practicable. At this point it should be noted that the objective of this test program was to evaluate the KCl polycrystalline material on a relative basis to the single crystal. Therefore a unified specimen design may well serve this purpose.

Figure 6 shows the specimen design used for all tests. The choice of this geometry was aimed at facilitating the cutting of the final notch. This notch was made with a jeweler's saw blade (0.01 inch thick) for tests #1 and #2 and a string saw (0.005 inch thick) for tests #3 through #10. Silicon carbide grit was used to facilitate cutting of the sharp notch which was then carefully cleaned with xylene followed by a rinse with pure methyl alcohol. The samples were stored in a dessicator until testing.

The Instron TM-L was used for three-point bending of these crystals. The applied load was then plotted against the cross head displacement. It is assumed that in view of the extreme notch sensitivity of the specimens and their brittle fracture behavior at room temperature, the crack opening displacement could be linearly related to the cross head displacement. The cross head speed was 0.002 inch/minute. In addition, this arrangement avoids complications which arise from the use of a displacement gauge together with glued-on knife edges at the notch sides.

K_{IC} is calculated by the expression

$$K_{IC} = \frac{P_2 S}{BW^{3/2}} f(\frac{a}{W})$$

where

P_2: load as determined in the ASTM E399-70T

S: span length = 1.5 inch for all tests

B: pecimen thickness in inches

W: depth of specimen in inches

a: crack length as determined in ASTM E399-70T

$$f(\frac{a}{W}) = [2.9(\frac{a}{W})^{1/2} - 4.6(\frac{a}{W})^{3/2} - 21.8(\frac{a}{W})^{5/2} - 37.6(\frac{a}{W})^{7/2} + 38.7(\frac{a}{W})^{9/2}]$$

Table II shows the calculated values of K_{IC} for single KCl and polycrystalline KCl. The averaged values of K_{IC} and G_{IC} are given in Table III where only tests #3 through 10 were averaged since the jeweler saw notches were inadequate in the first two tests. In addition, a special polishing jig was made to insure parallel upper and lower surfaces of the specimens #3 through 10. Although these tests are still qualitative, it is clear that the polycrystalline samples showed a definite improvement in K_{IC} values over the single crystals by a factor of about three and in G_{IC} values by a factor of twelve.

FRACTOGRAPHY

In order to determine the mechanism of fracture in these specimens and to shed some light on their microstructures, the fracture surfaces were examined in the scanning electron microscope. Figure 7a shows the crack initiation site in the single crystal with "cleavage fans" normal to the crack tip. Cleavage occurs along the {100} planes and may simultaneously take place on two or more planes resulting in the formation of steps as shown in Figure 7b. Figure 8a shows the topography of the fracture surface in a polycrystalline sample. As may be noted, the surface roughness is much greater than in the case of single-crystal fracture. The mechanism of fracture however appears to be the same, namely, cleavage along discrete planes within the differently oriented grains. Similar surface steps are shown on Figure 8b.

The fracture surface micrograph shown in Figure 9a clearly indicates the existence of certain microstructural units of the order of 5μm in size. By contrast, Figure 8a reveals some larger microstructural units whose sizes are of the order of 500 μm. Also Figure 9b shows little misorientation between the 5μm structural units and that the cleavage surface may extend over several such units without suffering sharp angular changes. Figure 9c shows a close-up on these units of structure. Their boundaries apparently have been etched by letting the crystals stand for half an hour in room air. "Grooving" of such boundaries may eventually lead to surface embrittlement of the polycrystals, a fact which points out the importance of protective coatings for the polycrystalline windows.

FLAW DETECTION

Simple machining tests (Figure 10) provide one possible means of studying the distribution of defects which cause fracture in polycrystalline KCl, provided that the chip characteristics are sensitive to the same defects (cracks) that cause tensile fracture. It was hoped that variation of rake angle in cut on a milling machine would show a minimum number of defects at a particular rake angle. At high rake angles, from 40° up, there is irregular brittle fracture with a typical roughness of 50μ. At a low rake angle of 30°, three mechanisms appear. Smooth flat surfaces prevail over the majority of the cut surface. Chunks of material of the order of 50μ and larger are pulled out at random intervals from the bottom surface and edges of the cut. In some cases small cracks are present that originate in the corners of the cut and propagate into the bulk of the material for short distances. The question that arises is whether these three distinct effects that appear at a 30° rake angle are a result of the gross mechanics of cutting or a result of original defects in the sample. Tensile fracture toughness testing of the same material has shown similar large chunks of material pulled out. The size of the fracture facets (≈50μ) are larger than the grain size (≈10μ) observed from etched surfaces.

Further tests are being made on the crystals using the milling machine with forces monitored by a solid state load cell. In order to reduce the apparent defects, both small increases in temperature and oblique cutting will be tried. Cooling-induced fracture tests are being planned to induce fracture on the surface of KCl utilizing a jet of liquid nitrogen or other coolant. It is proposed that this jet will induce fracture if the proper flow rate is applied. Control of the flow rate and speed across the surface will assure a continuous reproducible test. Once a particular set of conditions are established, large samples can be checked for defects in short time periods of straightforward testing.

WINDOW COATINGS

In order to provide protection against moisture attack and to allow the IR windows to be anti-reflective, thin surface coatings are required. It has already been noted in the section on the strength of polycrystalline KCl materials that the surface preparation and subsequent protection of a thin film can also dramatically improve the mechanical characteristics.

Studies have continued during this quarter on the preparation of polycrystalline KCl surfaces and of the deposition parameters to produce adhering, protective coatings. Germanium, magnesium fluoride and sodium fluoride have been deposited. The Ge coatings (<6800 Å) were deposited at room temperature by electron beam evaporation. The transmission spectrum was similar to results reported by Donovan et.al.[5] for Ge on KCl single crystals. The MgF_2 was also deposited in vacuum at room temperature. For the deposition of NaF, a flowing Ar/H_2 gas mixture was used to carry evaporated NaF (T=1000-1250°C) to a hot (200-300°C) substrate. The system is shown systematically in Figure 11. Since the thermal expansion coefficients of KCl and NaF are almost the same, deposition at high temperatures which provides good adhesion will not result in crazing or cracking of the coating upon cooling. The vapor pressure of NaF at the evaporation temperatures is 1-10 mm and yields sufficient transport rates to the cooler substrate for deposition times of about an hour. The short deposition time for micron thick coatings minimizes grain growth problems.

Initial tests of the stability of the Ge-coated polycrystalline KCl were performed with a pulsed CO_2 laser. The pulse duration was about 0.5 microsec. The energy of the beam was about 1 megawatt focused to an area of about 1/4 cm^2 of the Ge-coated surface. Figure 12(a) shows a region after two pulses and demonstrates that the Ge is easily vaporized and that subsequent bursts (up to 20 in Figure 12(b)) causes further cracking and spalling of the polycrystalline KCl at the surface (<200μ). The surface cracks are shown in the scanning electron micrograph (Figure 12(c)) as a further indication of the strong orientation effects which remain in the hot-worked material. The surface cracks show definite cubic morphology with a size larger than the observed grain size. The material is, nevertheless, resistant to catastropic failure in as much as the cracks do not readily propagate.

NATURE OF THE GRAIN BOUNDARIES

Observations have been reported in several sections of this report as to the degree of polycrystallinity and to the strengthening features of the grain boundaries of hot-worked KCl. The term "polycrystalline" has been used to describe the material and indeed etched micrographs of the hot-worked material show a typical polycrystalline network of etched boundaries. The increases in the mechanical properties appear to follow the predicted behavior due to grain boundaries; however, several experimental observations have been made which at first appear contradictory in light of a polycrystalline body with only two independent room temperature slip systems and with preferential (100) cleavage planes. A short summary of these observations is given here with the tentative conclusion as to the nature of the grain boundaries.

(1) The preliminary results of x-ray diffraction and electron microscopic

examination of a replicated boundary indicate grain to grain misorientations of a few degrees to about 20 degrees. The grain size from etched micrographs appears to be about 10μm.

(2) A strong textural relationship between the orientation of the hot-worked single crystal and the orientation of the grains within the polycrystalline body was noted from the microhardness measurements and Laue patterns. The polycrystalline yield and fracture strengths also show these textural effects.

(3) The stress-strain behavior of polycrystalline KCl that was carefully water polished and coated indicates that significant plastic flow within the grains and probably through the grain boundaries occurs before the work-hardened piece fractures. This is inconsistent with the notion that five independent slip systems are required if cracks are not to form at inpenetrable grain boundaries.

(4) The fractographs of single crystal KCl and polycrystalline KCl show similar features on a scale much larger than the grain size. Large grain-to-grain misorientation combined with the characteristic of preferred fracture on (100) planes should cause distinctly different fracture surfaces. The laser damage cracks also showed planar features over a size of hundreds of microns.

(5) The strength and plane strain fracture toughness are significantly increased over single crystalline values and the values caused from strain hardening alone[6]. This is consistent with the notion of a polycrystalline body.

From these several observations, it is assumed that the predominant textural effects of recrystallized hot-worked KCl results in an array of low angle grain boundaries which might more properly be called subgrain boundaries. More X-ray studies are planned in order to determine grain to grain misorientation. The presence of subgrains rather than highly misoriented grains may be of advantage in the laser window application because the ductility reduces the probability of catastropic failure due to mechanical or thermal loading.

DOUBLE SALT SINGLE CRYSTAL GROWTH

In the previous report, alkali halide - alkaline earth halide double salts were recommended as possible IR transmitting window materials. Single crystals of $BaCl_2 \cdot 2KCl$ were grown by the Bridgman method. Since the hardness of this material was found to be very high, 70-80 Kg/mm^2, the optical properties of this material were examined during the period covered in the present report. For this purpose, larger single crystals were grown by the Czochralski technique.

Crystal growth. Smaller crystals grown by the Bridgman method were used as the seed crystals to grow the larger crystals by the standard Czochralski method. The starting chemicals were the same as previously reported. The crystal growth was performed from a stoichiometric melt. Because of the continuous evaporation, the composition during the growth period might be slightly different. The chemicals were dried at 600°C over night under vacuum. Nitrogen gas was flowed over the melt during crystal growth. The pulling speed was ≈1 mm/hr.

The grown crystals had a volume of about 1 cubic inch. Cracks which developed during cooling could not be eliminated. This was probably because the piece consisted of several grains and because the crystal has an anisotropic thermal expansion coefficient.

IR transmission measurements. Samples (10 X 10 X 5 mm) were cut from the crystals and the surfaces polished with 1 micron alumina abrasive powder in kerosene. The surface prepared in the air was, however, opaque indicating a significant surface attack by water vapor.

The measurement was performed with a double beam Beckman 12 spectrometer. The spectrum is sketched in Figure 13 and is quite similar to alkali halides as predicted in the previous report. The IR cutoff occurs at about 14μ. The absorbance of this crystal in the wave length range of interest (≈11μ) was, however, high, $\beta = 1.7\ cm^{-1}$. This was probably due to the presence of crack within the specimen, poor surface finishing, and impurities. The hygroscopic nature of this material is indicated by the two strong absorption peaks at 6.1μm and 2.8-3.1μm, which correspond to the absorption of water. The total absorption can probably be reduced by better crystal growth conditions and sample preparation, but the lower limits of β cannot at present be predicted.

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LIST OF INVESTIGATORS

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Professor W.A. Backofen	Dr. K. Kitazawa
Professor H.K. Bowen	S.M. El-Soudani
Professor R.L. Coble	C. Lemaignan
Professor F.A. McClintock	B.R. Leseur
Professor R.M. Pelloux	J.R. Mathews
Dr. R. Singh	M.F. Yan

Table I

Vickers Hardness
(25 gm load)

Polycrystalline KCl (100)	10.3 kg/mm^2
Polycrystalline KCl (110)	15.5 kg/mm^2
Polycrystalline KCl (111)	12.5 kg/mm^2
Polycrystalline $KCl_{.33}Br_{.67}$ (100)	21.4 kg/mm^2
$BaCl_2 \cdot 2KCl$ Crystal	70-80 kg/mm^2

Table II

Plane Strain Fracture Toughness of Single Crystals and Polycrystals
of KCl as Determined by Three-Point Bending

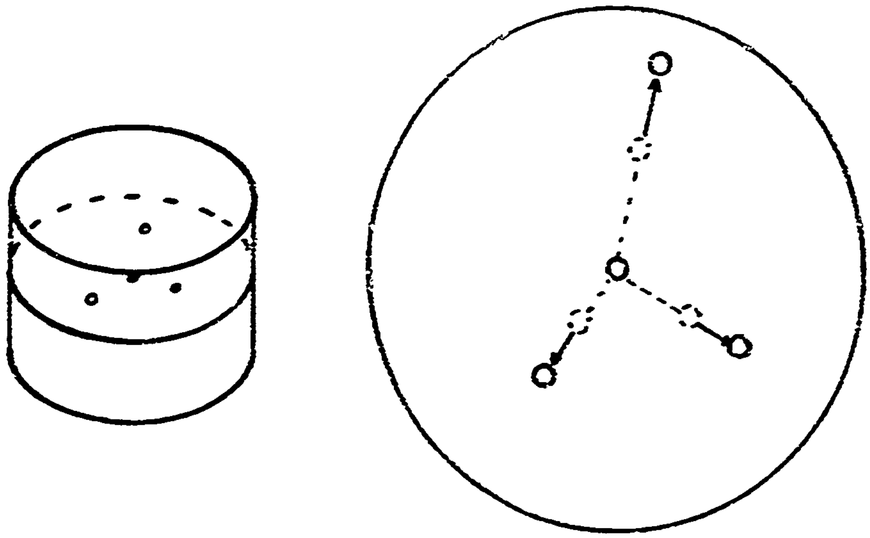
Test Number	Final Notch	K_{IC} [psi $\sqrt{inch}$]	
		Single Crystals (Crack Plane {100})	Polycrystals (Hot-worked Along <100>)
1	Jeweler's Saw	655	-
2	0.01" thick	-	700
3	String Saw	298	-
4	0.005" thick	324.4	-
5	"	230.6	-
6	"	322.5	-
7	"	232.3	-
8	"	224.3	-
9	"	-	855
10	"	-	1030

Table III

Comparative Evaluation of KCl
Single and Polycrystals

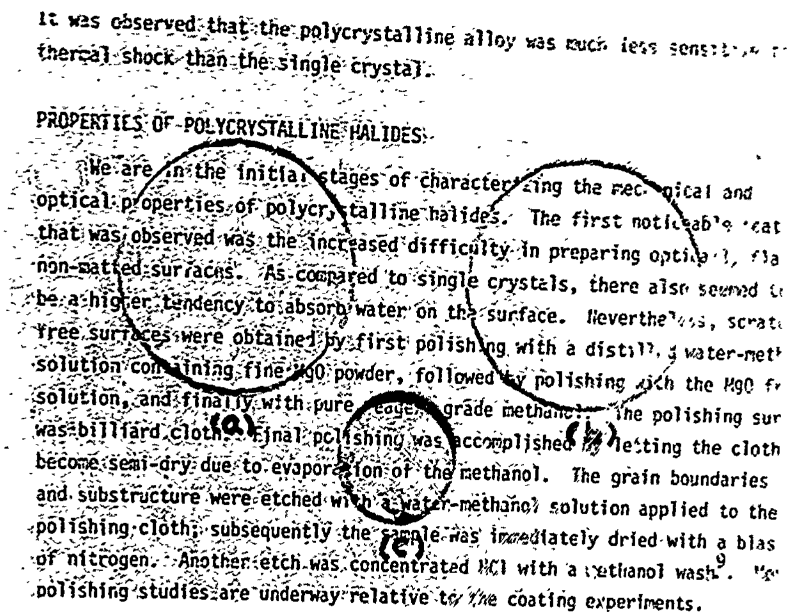
Property	Single Crystal (Crack Plane {100})	Polycrystals (G.S. =10μ) (Deformed Along <100>)	Property Ratio $\left(\frac{\text{Polycrystal}}{\text{Single Crystal}}\right)$
K_{IC} psi $\sqrt{\text{inch}}$	272	942.5	3.45
G_{IC} erg/cm^2	3020	36200	12

Estimated critical plastic zone size, $\frac{1}{2\pi}\left(\frac{K_{IC}}{\sigma_{yield}}\right)^2$, is 0.13 in.for single crystal KCl and 0.038 in.for polycrystalline KCl.



(a) (b)

Figure 1 - Schematic representation of experiment to determine flow properties of hot-worked KCl crystals. (a) The 4 holes drilled into one face of a cleaved crystal were carried with the mass flow outward along radial lines of direction (b).



It was observed that the polycrystalline alloy was much less sensitive to thermal shock than the single crystal.

PROPERTIES OF POLYCRYSTALLINE HALIDES

We are in the initial stages of characterizing the mechanical and optical properties of polycrystalline halides. The first noticeable feat that was observed was the increased difficulty in preparing optical, fla non-matted surfaces. As compared to single crystals, there also seemed to be a higher tendency to absorb water on the surface. Nevertheless, scrat free surfaces were obtained by first polishing with a distilled water-meth solution containing fine MgO powder, followed by polishing with the MgO fr solution, and finally with pure reagent grade methanol. The polishing sur was billiard cloth. Final polishing was accomplished by letting the cloth become semi-dry due to evaporation of the methanol. The grain boundaries and substructure were etched with a water-methanol solution applied to the polishing cloth; subsequently the sample was immediately dried with a blas of nitrogen. Another etch was concentrated HCl with a methanol wash[9]. Mo polishing studies are underway relative to the coating experiments.

Figure 2 - Hot-worked, polycrystalline halide windows. (a) 2 inch KCl (100) orientation, (b) 2 inch KCl (110) orientation, (c) 1 inch $KCl_{.33}Br_{.67}$ (100) orientation.

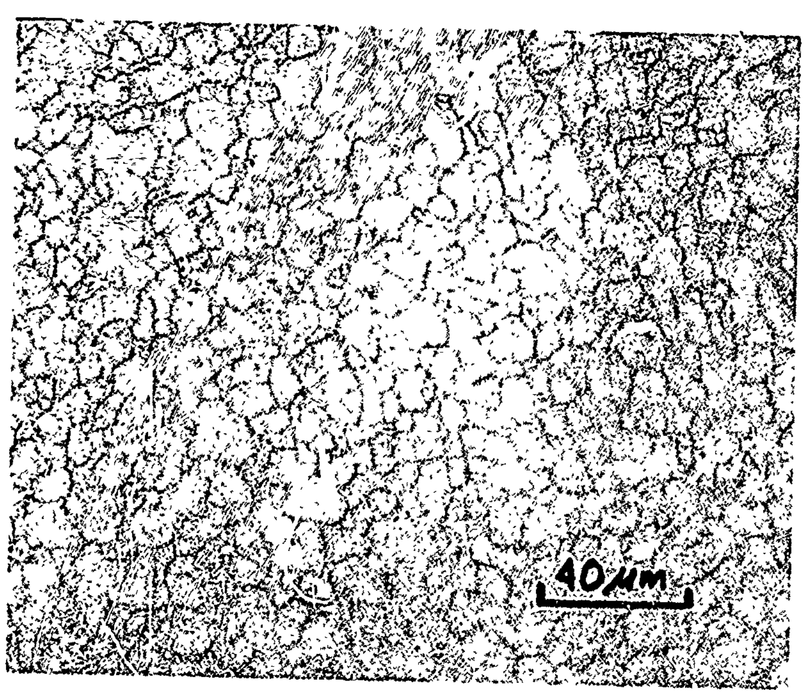


Figure 3 - Etched photomicrograph of hot-worked KCl crystal with (110) orientation.

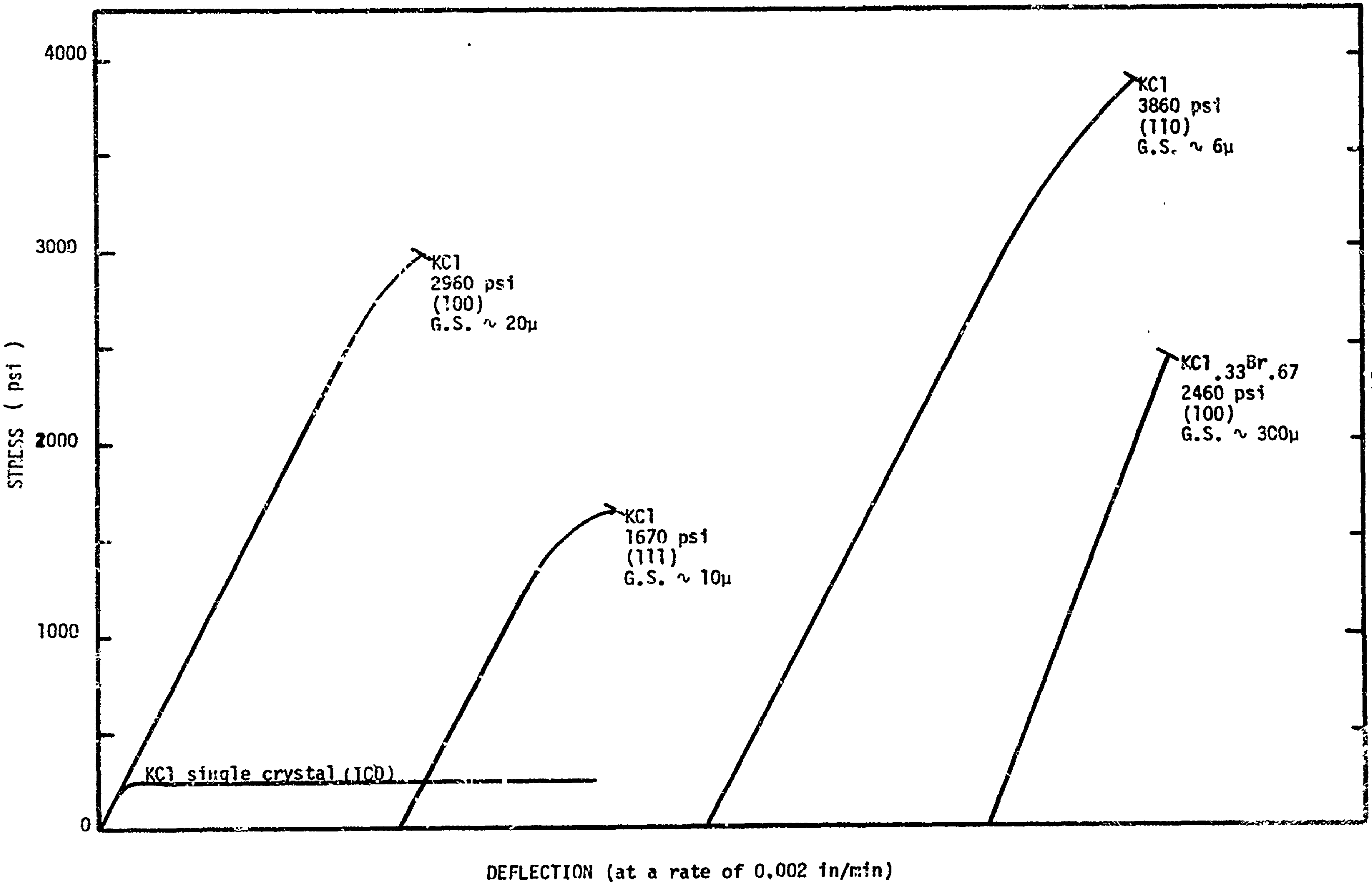


Figure 4 - Stress-Deflection curves for polycrystalline halides.

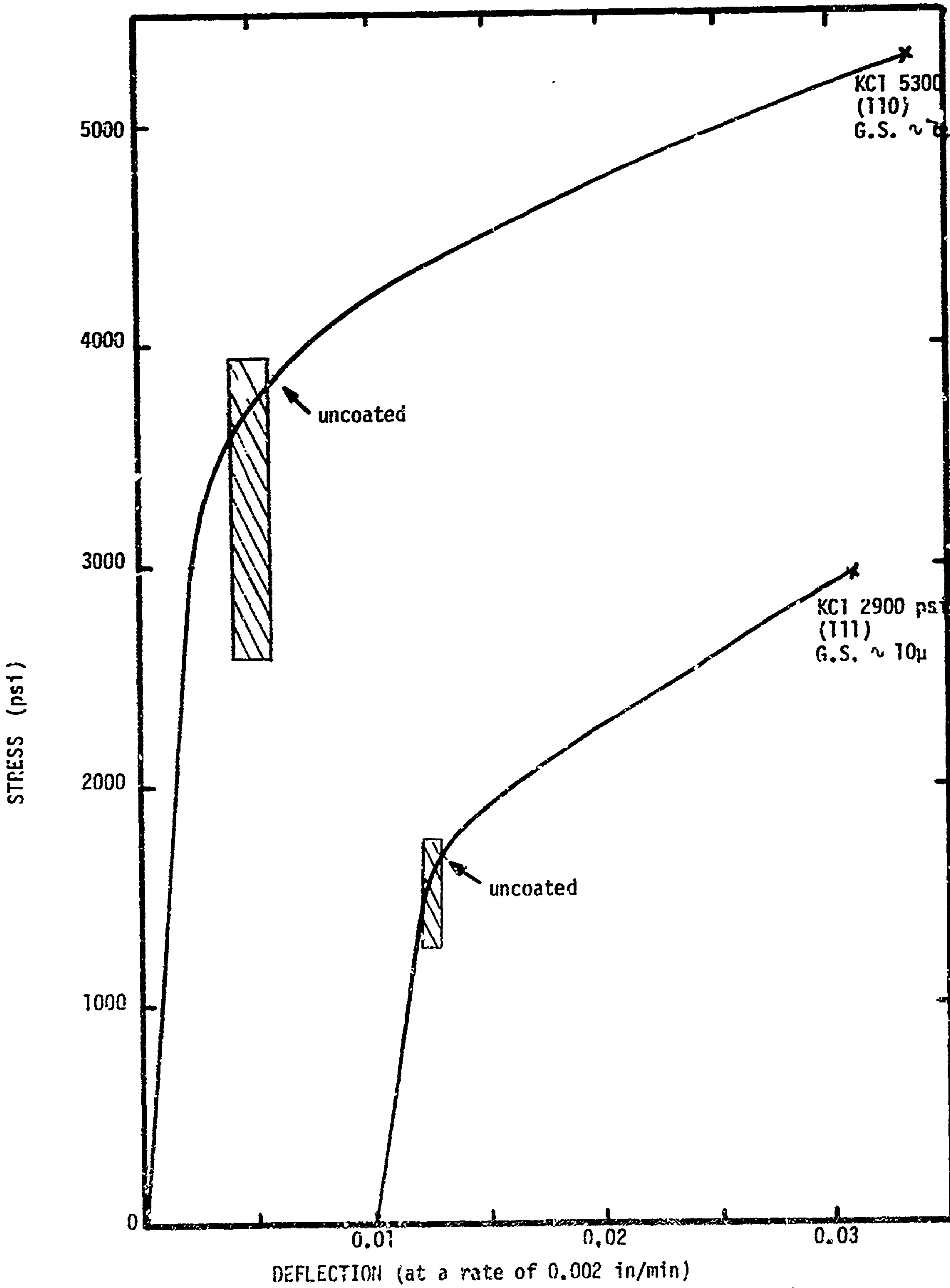


Figure 5 - Stress-Deflection curves for polycrystalline KCl samples with water etched surface and lacquer coatings.

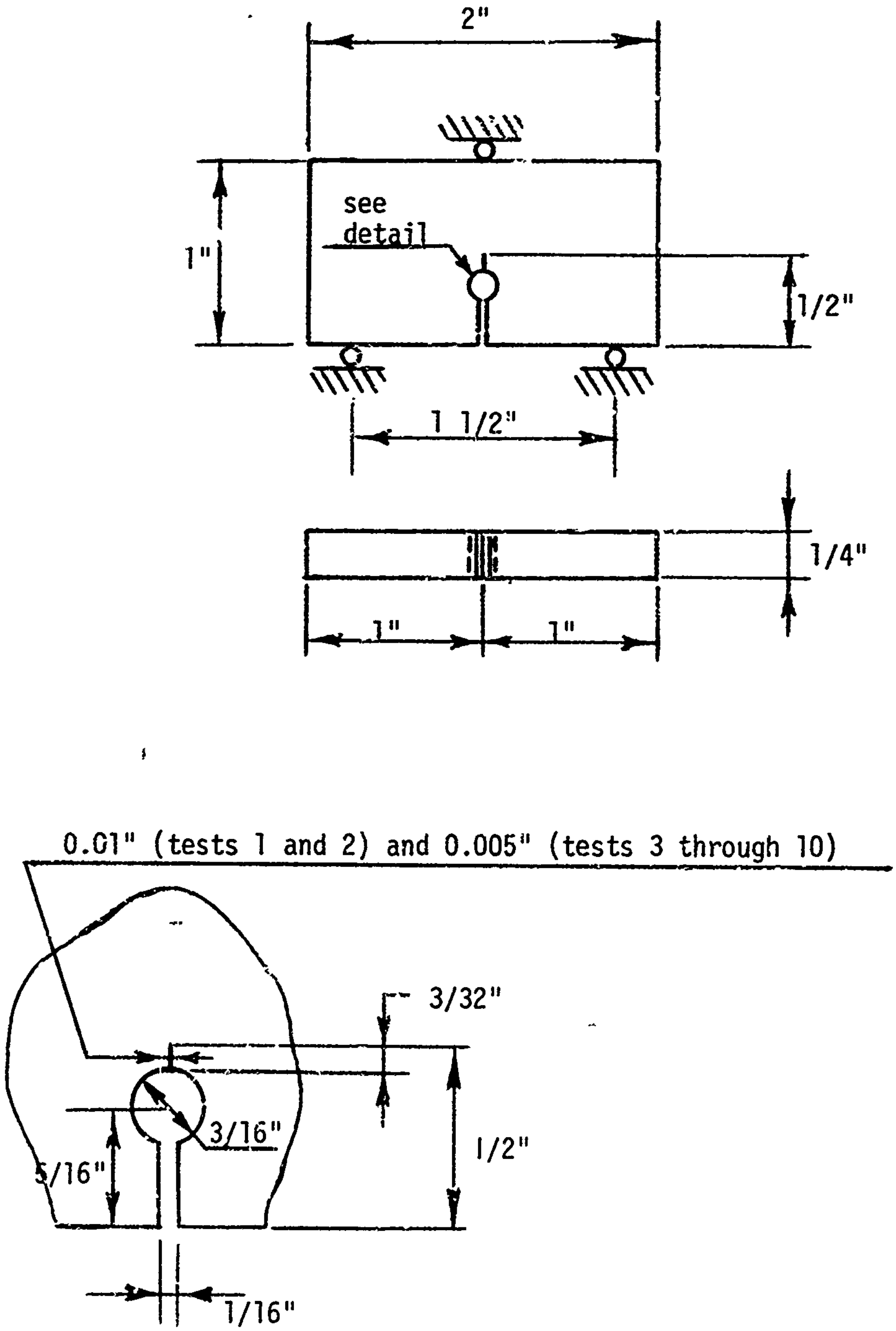
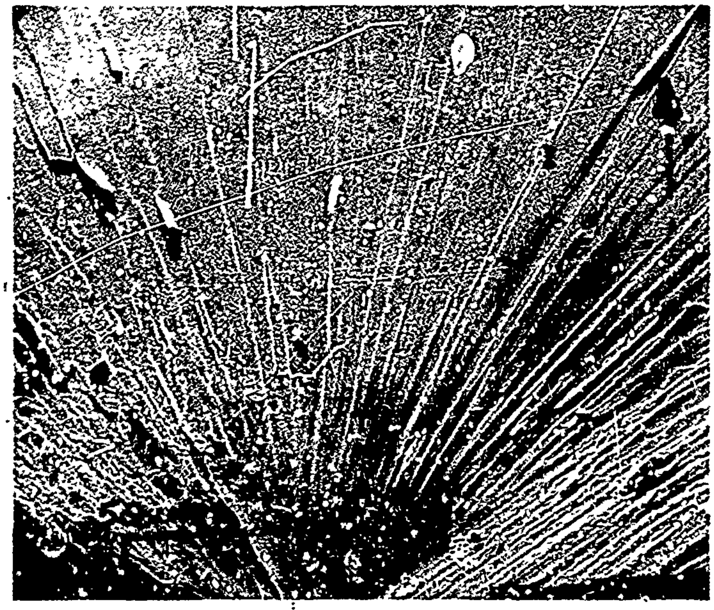
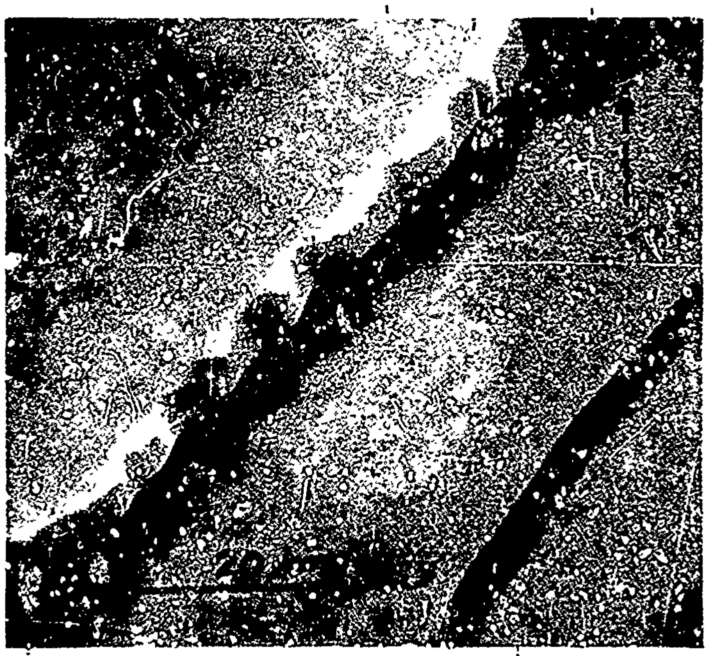


Figure 6 - Sample geometry for plane strain fracture toughness tests.

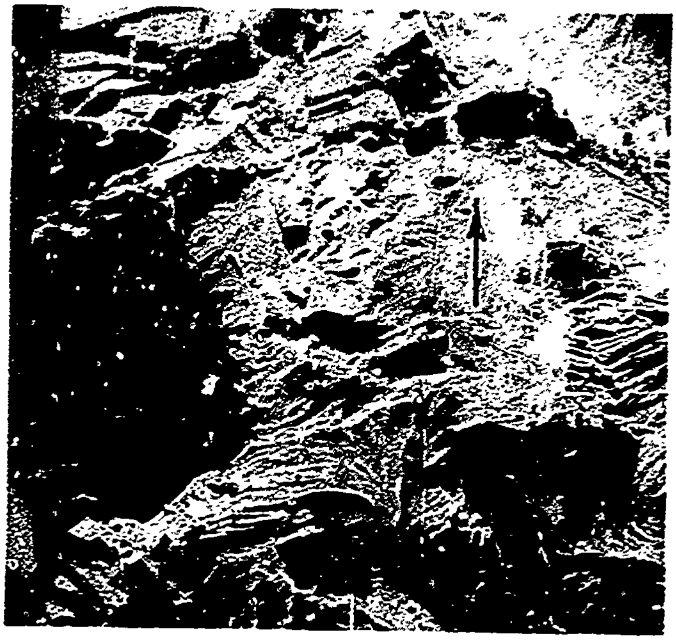


(a)



(b)

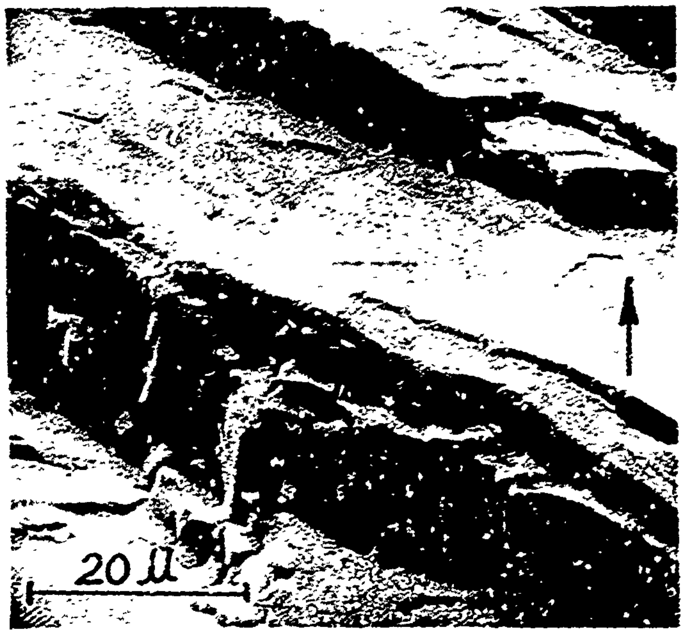
Figure 7 - Fracture surface of KCl single crystal (fracture plane is (100)).



(a)

(b)

Figure 8 - Fracture surface of polycrystalline KCl. (Specimen was cut from hot-worked KCl crystal deformed along <100> direction.)



(a)

(b)

Figure 9 - Fracture surfaces of polycrystalline KCl showing etched "grain boundaries".

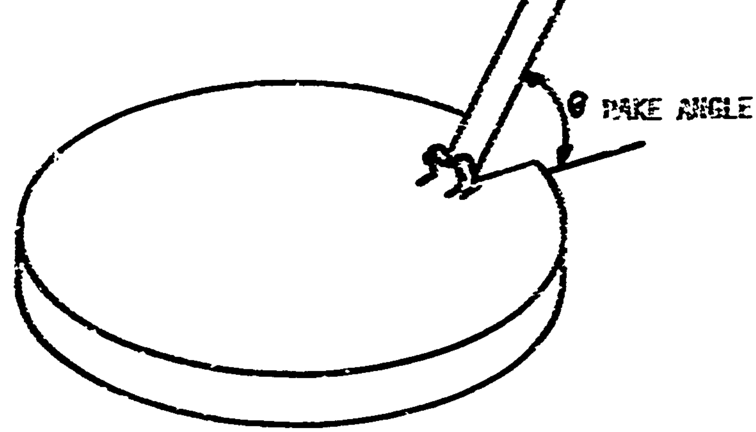


Figure 10 - Flaw detection in polycrystalline KCl.

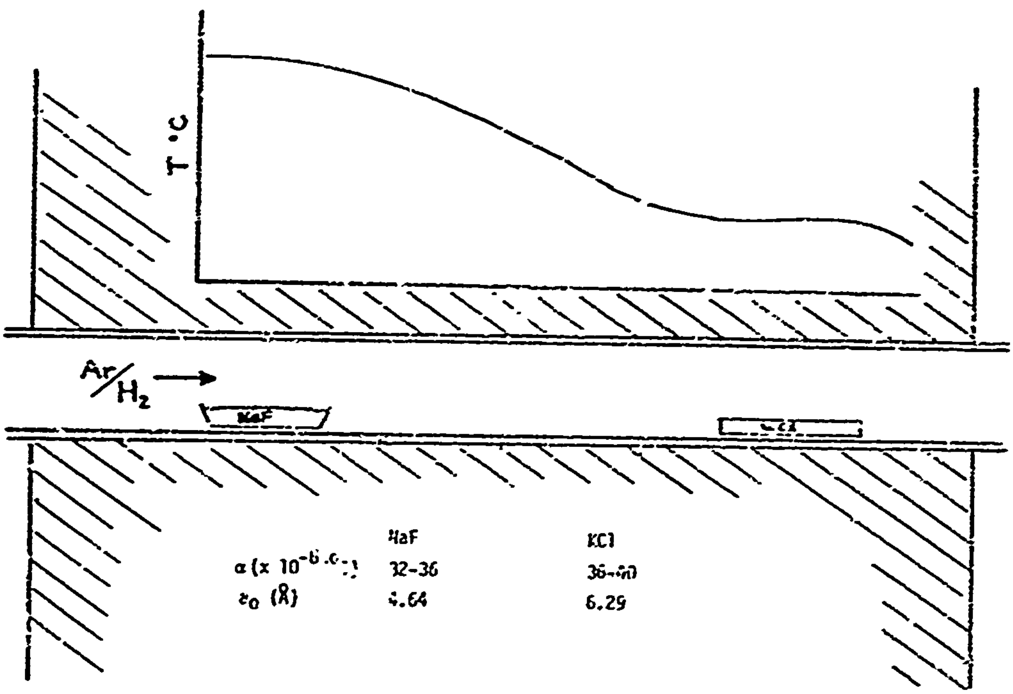


Figure 11 - Vapor transport of NaF onto polycrystalline KCl.

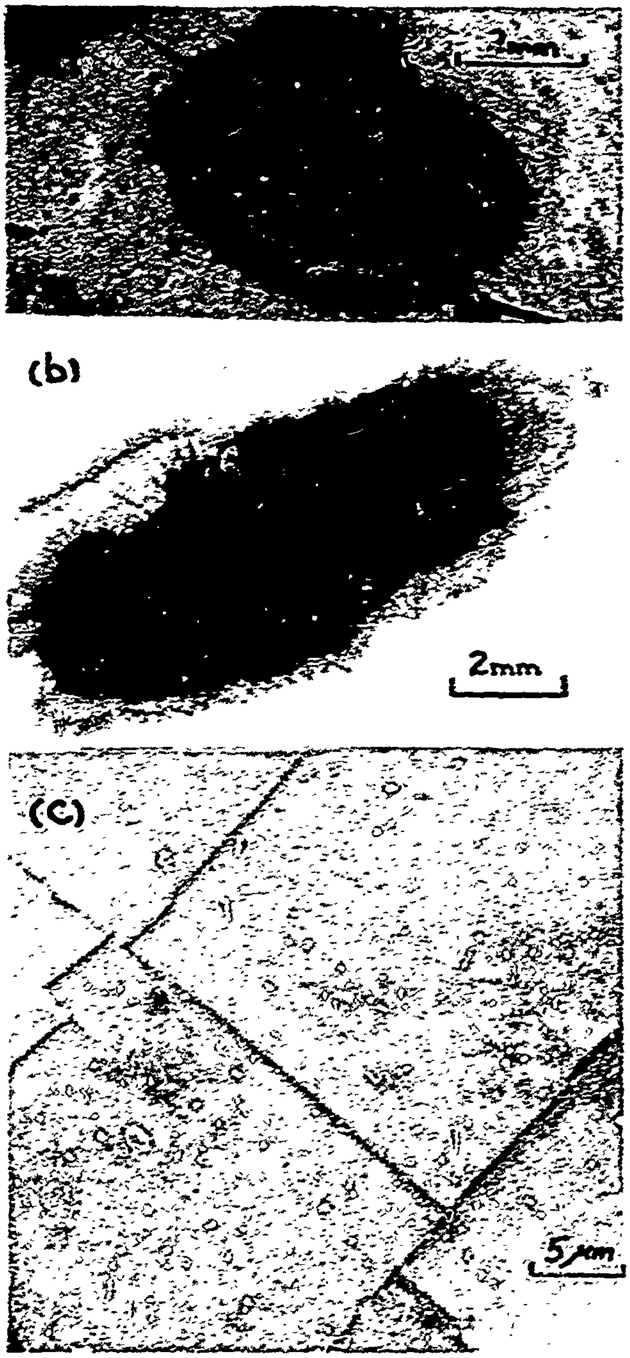


Figure 12 - Damage to Ge-coated polycrystalline KCl from 1 megawatt CO_2 pulsed laser (a) vaporization of Ge after 2 pulses, (b) further surface spallation after 20 pulses, (c) scanning electron micrograph of surface cracks.

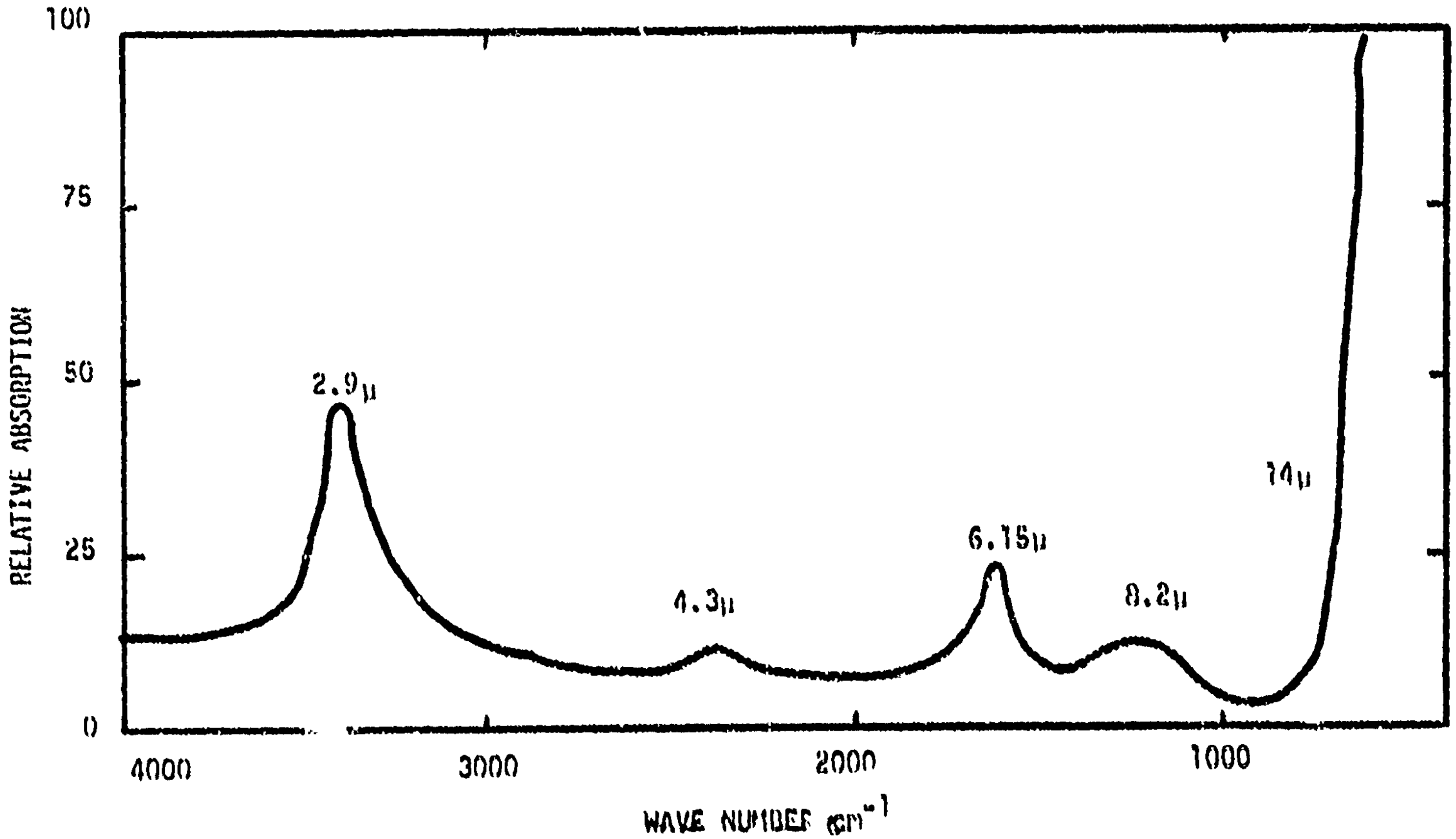


Figure 13 - IR absorption spectrum of $BaCl_2 \cdot 2KCl$ crystal.